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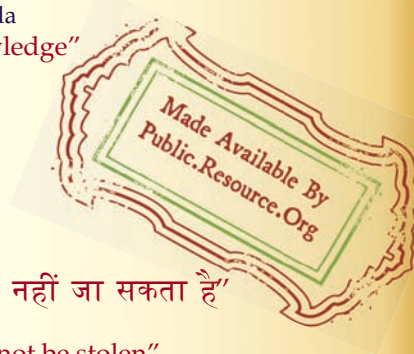
IS 15752 (2007): Sericite for ceramic industry [CHD 9: Ceramicware]



“ज्ञान से एक नये भारत का निर्माण”

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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
सिरैमिक उद्योग के लिए सेरीसाइट — विशिष्टि

Indian Standard
SERICITE FOR CERAMIC INDUSTRY —
SPECIFICATION

ICS 73.060.01; 81.06.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

A micaceous-type mineral with composition indicating that it probably originates from the alteration of feldspar. Sericite is a hydrous aluminosilicate of potassium [$K_2Al_6Si_6O_{22}(OH)_4$ or $K_2O \cdot 3Al_2O_3 \cdot 6SiO_2 \cdot 2H_2O$] with 11.83 percent K_2O , 38.40 percent Al_2O_3 , 45.25 percent SiO_2 and 4.52 percent H_2O . It is a rare mineral found generally with pyrophyllite in metamorphic rocks. It has properties similar to talc having distinct greasy feel. It has a pearly lustre and white, light green, grey or brown in colour depending upon the impurities present.

It is widely used in earthenwares, glazed tiles and porcelain insulators. Their shrinkage on firing is small and the product is fairly resistant to thermal shock by virtue of its high thermal conductivity and low coefficient of thermal expansion.

This standard contains **5.2**, **5.3** and **6.1** which provide for agreement between the purchaser and the supplier.

The composition of the Committee responsible for formulation of this standard is given in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SERICITE FOR CERAMIC INDUSTRY — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sericite suitable for use in ceramic industry.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 1993	Hydrochloric acid — Specification (<i>fourth revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
799 : 1985	Specification for ammonia, liquor (<i>second revision</i>)
915 : 2006	One-mark volumetric flasks — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
1997 : 1982	Specification for burettes (<i>second revision</i>)
2781 : 2004	Glossary of terms relating to ceramicware (<i>second revision</i>)
4905 : 1968	Methods for random sampling
8883 (Part 1) : 2005	Methods of sampling chemical and chemical product: Part 1 General requirements and precautions (<i>first revision</i>)
10332 : 1982	Specification for hydrofluoric acid, aqueous

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2781 shall apply.

4 GRADES

There shall be two grades of material, namely:

- a) *Grade 1* — Suitable for low maturing crockery, sanitarywares and vitreous wares.
- b) *Grade 2* — Suitable for whiteware and insulator industry.

5 REQUIREMENTS

5.1 Description

The material shall be in the form of powder or lumps prepared from naturally occurring sericite rocks. It shall be free from foreign matter.

5.2 Moisture Content

Unless otherwise agreed to between the purchaser and the supplier, the moisture content, as received shall not exceed 2 percent by mass, when determined by the method prescribed in Annex A.

5.3 Size Grading

The grain sizes of Grade 1 and Grade 2 material shall be as agreed to between the purchaser and the supplier.

NOTE — Normally, for ceramic industry the grain size is finer than 150 microns. However, the lumps may also be supplied.

5.4 Relative Density

The relative density of the material shall be between 2.8 and 2.9 when tested in accordance with the procedure prescribed in Annex B.

5.5 Fusibility

5.5.1 General

The fusion characteristics of sericite is expressed in terms of Orton Standard Pyrometric Cone Equivalent (PCE).

5.5.1.1 The PCE for Grade 1 shall be Orton Standard Cone No. 8 (1 300°C) and for Grade 2 Orton Standard Cone No. 15 (1 430°C) when determined in accordance with the method prescribed in Annex C.

5.5.2 Fired Colour

The colour of fired sericite shall be dull white to cream for both the grades.

5.6 Chemical Composition

Sericite shall also comply with the requirements prescribed in Table 1 when tested in accordance with the method prescribed in Annex D. Reference to the relevant clauses of Annex D is given in col 5 of Table 1.

Table 1 Requirements of Sericite for Ceramic Industry

Sl No.	Characteristics	Requirements		Methods of Test, Ref to Annex
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Loss on ignition, percent by mass, <i>Max</i>	6.0	7.0	D-2
ii)	Silica (as SiO ₂), percent by mass, <i>Min</i>	45.0	50.0	D-3
iii)	Alumina (as Al ₂ O ₃), percent by mass, <i>Min</i>	33.0	32.0	D-4
iv)	Iron oxide as (Fe ₂ O ₃) ¹⁾ , percent by mass, <i>Max</i>	1.0	1.5	D-5
v)	Titanium dioxide as (TiO ₂) ¹⁾ , percent by mass, <i>Max</i>	1.0	1.0	D-6
vi)	Calcium oxide (as CaO), percent by mass, <i>Max</i>	1.0	1.5	D-7
vii)	Potassium oxide (as K ₂ O) and sodium oxide (as Na ₂ O), percent by mass, <i>Min</i>	7.0	5.0	D-8

¹⁾ Sum of percent of iron oxide (as Fe₂O₃) and titanium oxide (as TiO₂) for Grade 1 and Grade 2 shall not exceed 1.5 and 2.0 percent respectively.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in jute bags or as agreed to between the purchaser and the supplier.

6.2 Marking

6.2.1 Each bag shall be marked with the following:

- Name of the manufacturer or his recognized trade-mark, if any;
- Name and grade of material;
- Net mass of content;
- Grain size;
- Lot number or batch number in code or otherwise; and
- Month and year of manufacture.

6.2.2 BIS Certification Marking

The bags may also be marked with the Standard Mark.

6.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING AND CRITERIA FOR CONFORMITY

7.1 The representative sample shall be drawn as prescribed in Annex E.

7.2 Test Sample

Grind 5 to 10 g of the sample in a porcelain mortar so that it passes through 75 micron sieve. Dry to a constant mass at 105 ± 2°C and use it for the purpose of chemical analysis.

7.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this standard, if all the test results meet the relevant requirements.

ANNEX A

(Clause 5.2)

DETERMINATION OF MOISTURE CONTENT

A-0 PROCEDURE

Weigh accurately nearest to 0.01 g, about 20 g of the sample in a tared dish and dry at 105 to 110°C in an air-oven for 30 min. Cool in a desiccator and weigh. Repeat heating and cooling till constant mass is obtained.

A-1 CALCULATION

$$\text{Moisture content, percent by mass} = \frac{M - M_1}{M} \times 100$$

where

M = mass of the sample taken for the test, in g; and

M_1 = mass of the sample after drying, in g.

ANNEX B

(Clause 5.4)

DETERMINATION OF RELATIVE DENSITY

B-0 APPARATUS

B-0.1 Analytical Balance

B-0.2 Pyknometer — 50 ml capacity with a ground capillary stopper.

B-0.3 Vacuum Source — To produce a vacuum of 12 to 25 mm of mercury.

B-1 PROCEDURE

B-1.1 The pyknometer shall be dried at room temperature after rinsing it with acetone or any quick drying solvent and then with hot air. Weigh the pyknometer and record the mass as M . Fill it with distilled water at room temperature and note the room temperature. Insert the stopper and wipe the excess water from the tip in such a manner as not to withdraw any water from the capillary. Thoroughly dry any water adhering to the outside of the pyknometer and weigh recording the mass as M_1 .

B-1.2 Empty the pyknometer and dry it again. Introduce about 10 g of the dried sample and weigh with the stopper in position, recording the mass as M_2 . Remove the stopper, fill the pyknometer with distilled water to one-fourth to one-half its capacity and keep the pyknometer in a vacuum desiccator and apply

vacuum to remove the entrapped air completely. Release the vacuum and take out the pyknometer from the desiccator and keep it under room temperature for few minutes to attain normalcy.

B-1.3 Fill the pyknometer with water. Insert the stopper, wipe the excess water from the tip and the outside of the pyknometer as above and weigh recording the mass as M_3 .

B-2 CALCULATION

B-2.1 Calculate the relative density as follows:

$$\text{Relative density, } t^\circ/27^\circ = \frac{M_2 - M}{(M_2 - M) - (M_3 - M_1)} \times d$$

where

t = temperature of the water, in $^\circ\text{C}$;

M = mass of empty stoppered pyknometer, in g;

M_1 = mass of stoppered pyknometer filled with water, in g;

M_2 = mass of stoppered pyknometer and sample, in g;

M_3 = mass of stoppered pyknometer with sample and water, in g; and

d = density of water at 27°C .

ANNEX C

(Clause 5.5.1.1)

DETERMINATION OF FUSIBILITY

C-0 GENERAL

The object of this test is to determine the softening point of sericite by comparing the test cones prepared from the material under test with standard pyrometric cones heated in a suitable furnace at a definite rate of rise of temperature.

C-1 APPARATUS

C-1.1 Furnace — Electrically fired furnace, capable of maintaining a natural or oxidizing atmosphere.

C-2 PREPARATION OF TEST CONES

Take about 100 g of the dry sample and grind to pass through 150 micron sieve, avoiding excessive reduction of size. Mix thoroughly with sufficient alkali-free dextrin, glue or starch solution in water and form into test cones in a metal mould, in the shape of tetrahedron measuring 8 mm on the sides of the base and 25 mm high. Dry these cones slowly.

C-3 PROCEDURE

C-3.1 Mount the test cones along with orton standard

pyrometric cones covering the appropriate range (see 5.5.1.1), around the outer edge of high alumina plaque (plaque made of 90 percent alumina and 10 percent china clay mixture) so that their bases are embedded approximately 3 mm at deep, and one of the faces is inclined towards the centre of the plaque at an angle of 82° with the horizontal.

C-3.2 Carry out heating of the mounted cones in the furnace at a uniform rate of 150°C/h until a temperature

of 1 000°C is reached. Then reduce this rate to 120°C per hour and continue heating till the deformation takes place. Avoid reducing conditions in the furnace during heating operation. Softening of the cones is indicated by the top bending over and the tip of the cone touching the plaque surface.

C-3.3 Report fusibility of the material in terms of that of an orton standard pyrometric cone which most nearly corresponds in time of complete bending the test cone.

ANNEX D

(Clause 5.6)

CHEMICAL ANALYSIS OF SERICITE

D-0 SAMPLING AND PREPARATION OF TEST SAMPLE

The material shall be sampled and prepared for chemical analysis as given in 7.1 and 7.2.

D-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

D-2 DETERMINATION OF LOSS ON IGNITION

D-2.1 Transfer 1 g of the test sample to a weighed (tared) crucible and introduce it into a muffle furnace and slowly raise the temperature to 950 to 1 000°C. After about 30 min, remove the crucible and cool in a desiccator and weigh. Repeat ignition and cooling to obtain constant mass.

D-2.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = loss in mass on ignition, in g, and

M = mass of the sample taken for the test, in g.

D-3 DETERMINATION OF SILICA

D-3.1 Outline of the Method

The sample is decomposed by fusion with sodium carbonate and extracted with acid. Silica is then determined gravimetrically by dehydration and baking

followed by hydrofluorization. The residual silica in the filtrate is determined photometrically using molybdenum blue method.

D-3.2 Reagents

D-3.2.1 *Sodium Carbonate* — Anhydrous.

D-3.2.2 *Dilute Hydrochloric Acid* (1:1), (1:3) and (1:19) (v/v) — see IS 265.

D-3.2.3 *Dilute Sulphuric Acid* (1:1) (v/v) — see IS 266.

D-3.2.4 *Hydrofluoric Acid* — 40 percent (see IS 10332).

D-3.2.5 *Potassium Pyrosulphate* — Solid.

D-3.2.6 *Ethylene Diamine Tetraacetic Acid (EDTA) Solution* — 0.025 N approximately. Dissolve 9.31 g of disodium salt of ethylene diamine tetracetic acid, dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$) in water and dilute to 1 litre.

D-3.2.7 *Phenolphthalein Solution* — Dissolve 1 g of phenolphthalein in 100 ml of rectified spirit.

D-3.2.8 *Dilute Ammonia Solution* (1:3) (v/v) — see IS 799.

D-3.2.9 *Ferric Ammonium Sulphate Solution* — Dissolve 40.82 g of ferric ammonium sulphate [$NH_4Fe(SO_4)_2 \cdot 12H_2O$] in water containing 80 ml of dilute hydrochloric acid (1:1) and dilute to 500 ml in a volumetric flask.

D-3.2.10 *Ammonium Molybdate Solution* — Dissolve 100 g of ammonium molybdate in water containing a few drops of dilute ammonia, filter if necessary, and dilute to 1 litre.

D-3.2.11 Oxalic Acid Solution — Dissolve 100 g of oxalic acid in water and dilute to 1 litre.

D-3.2.12 Ascorbic Acid Solution — 10 percent (m/m). Dissolve 10 g of the acid in 100 ml of water and filter, if necessary. Preserve in an amber coloured bottle. The solution, thus preserved may be used within 5 days.

D-3.2.13 Standard Silica Solution — 0.04 mg/ml.

- a) Fuse 0.200 g of pure silica (99.8 percent) in 5 g of sodium carbonate in a platinum crucible. Dissolve in 300 ml of water containing 8 to 10 g of sodium hydroxide, using a polyethylene beaker. Cool and dilute with water in a volumetric flask to 500 ml. Store in a polyethylene bottle; and
- b) Prepare immediately before use a standard solution containing 0.04 mg/ml by diluting the above stock solution ten times.

D-3.3 Procedure

D-3.3.1 Decomposition of the Sample

Weigh accurately about 0.5 g of the finely ground and dried (105 to 110°C) sample into a platinum crucible. Add 3 g of sodium carbonate; after mixing intimately, cover the dish with lid and heat over a Meker burner, slowly increasing the temperature with caution till the mixture fuses. Keep at this temperature for 10 min and finally heat till a clear melt is obtained. Remove from the burner and cool. Extract the melt in a porcelain dish first with water and then with (1:1) dilute hydrochloric acid.

D-3.3.1.1 Add about 30 ml of (1:1) dilute hydrochloric acid and 0.5 ml of dilute sulphuric acid. Cover the porcelain dish and allow to stand on a steam bath until disintegration of the melt is complete. Crush any large flakes with flattened end of a glass rod.

D-3.3.2 Gravimetric Determination of Insoluble Silica

Evaporate the solution in the porcelain dish to dryness and allow it to remain on the steam bath for another hour. Transfer the dish to an air oven maintained between 105 to 110°C and bake for 1 h. Cool the dish and add 20 ml of (1:1) dilute hydrochloric acid followed by 30 ml of hot water. Stir the mixture till solution of soluble salts appear to be complete. Digest the mixture on a steam bath for 10 min without stirring. Filter through No.42 filter paper and rinse the dish with (1:19) dilute hydrochloric acid, scrubbing the basin with a rubber-tipped glass rod. Wash the precipitate five times with (1:19) and hot dilute hydrochloric acid and then with hot water until free from chloride. Reserve the filtrate and washings for photometric determination of residual silica.

D-3.3.2.1 Transfer both the filter paper and precipitate to a tared platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the muffle furnace at 1 050°C to constant mass (1 h being normally sufficient). Cool in a desiccator and weigh recording the mass as M_1 . Moisten the contents of the cold crucible with water, add five drops of dilute sulphuric acid and 10 ml of hydrofluoric acid. Evaporate to dryness on a hot plate or sand bath, in a fume cupboard, taking care to avoid spurling.

D-3.3.2.2 Heat the crucible and residue, cautiously at first, over a gas flame until the disappearance of white fumes and finally for 5 min in the furnace at 1 050°C, cool in a desiccator and weigh recording the mass as M_2 .

NOTE — If the residual mass is more than 10 mg, repeat the treatment with dilute sulphuric acid and hydrofluoric acid, ignite cool, and weigh (as M_3). Then fuse the residue with 0.5 g of anhydrous sodium carbonate, dissolve in 10 ml of (1:4) dilute hydrochloric acid and transfer quantitatively to the filtrate and washings reserved in D-3.3.2.

D-3.3.3 Test Blank Solution

Prepare a test blank solution using the same procedure and quantities of reagents as detailed from D-3.3.1 to D-3.3.2 but excluding the sample and dilute up to the mark with water in a 250 ml volumetric flask.

D-3.3.4 Photometric Determination of the Residual Silica

Boil the filtrate and washings reserved in D-3.3.2 and residue to about 200 ml. Transfer to a 250 ml volumetric flask and make up to the mark with water.

D-3.3.4.1 Transfer 10 ml aliquot of the solution prepared in D-3.3.4 to a 50 ml volumetric flask. Also transfer 10 ml each of the test blank solution prepared in D-3.3.3. to six 50 ml volumetric flasks: Add to the six flasks 0, 1, 2, 3, 4 and 5 ml respectively of the standard silica solution. Then to all the flasks add 5 ml of EDTA solution, a drop of phenolphthalein solution and then dilute ammonia solution dropwise until the colour, changes to red. Then add 5 ml of ferric ammonium sulphate solution and dilute to about 30 ml with water (the pH of the solutions should be 1 to 1.5).

D-3.3.4.2 Add 5 ml of ammonium molybdate solution and allow to stand for 10 min. Then add 5 ml of oxalic acid solution followed, immediately by 5 ml of ascorbic acid solution and dilute the volume to 50 ml. Allow the solution to stand for 30 min for development of full colour.

D-3.3.4.3 Starting with the blank ('0' ml) solution transfer a suitable portion of the coloured solution one at a time to an absorption cell, record the photometric

readings using the light band centred approximately at 810 nm. Prepare the calibration curve from these readings against concentration of silica in mg (as SiO_2).

D-3.3.4.4 Similarly, transfer a suitable aliquot of coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of silica by means of the calibration curve and calculate the silica content in grams in 250 ml of solution prepared in **D-3.3.3**.

D-3.4 Calculation

$$\text{Silica, percent by mass} = \frac{M_1 - M_2 + M_3}{M} \times 100$$

where

M = mass of the sample taken for the test in **D-3.3.1**, in g;

M_1 = mass of crucible and contents before hydrofluoric acid treatment, in g;

M_2 = mass of crucible and residue after hydrofluoric acid treatment, in g; and

M_3 = mass of the silica present in 250 ml of solution prepared in **D-3.3.3**, in g.

D-4 DETERMINATION OF ALUMINA

D-4.1 Outline of the Method

Aluminium is separated from aliquot of solution prepared in **D-3.3.3** as sodium aluminate by treatment with sodium hydroxide and is complexed quantitatively with EDTA by boiling with excess of the reagent. The excess EDTA is back titrated with standard zinc solution at pH about 5.2 using xylenol orange indicator. The aluminium EDTA complex on boiling with ammonium fluoride liberates EDTA which when titrated with standard zinc solution gives the amount of aluminium present in the solution.

D-4.2 Reagents

D-4.2.1 Dilute Sulphuric Acid (1:1) (v/v) — see IS 266.

D-4.2.2 Hydrofluoric Acid — 40 percent (see IS 10332).

D-4.2.3 Sodium Hydroxide Solution — 10 percent (m/v).

D-4.2.4 Sodium Carbonate — Solid.

D-4.2.5 Dilute Hydrochloric Acid (1:1) (v/v) — see IS 265.

D-4.2.6 Ethylene Diamine Tetraacetic Acid (EDTA) Solution (0.01 M) — Transfer 100 ml of the solution prepared in **D-3.2.6** to a 250 ml volumetric flask and dilute with water up to the mark.

D-4.2.7 Methyl Orange — Dissolve 0.05 g of methyl orange in 100 ml of water.

D-4.2.8 Dilute Ammonia (1:5) — Dilute 25 ml of concentrated ammonia (rd 0.90) to 150 ml.

D-4.2.9 Acetate Buffer (pH 5.2) — Dissolve 21.5 g of sodium acetate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

D-4.2.10 Standard Aluminium Solution — Weigh 0.5293 g of aluminium metal (99.9 percent purity) into a beaker containing 40 ml of dilute hydrochloric acid (1:1) and small drops of mercury. Heat the beaker over a water bath. Transfer the solution to a 1 000 ml volumetric flask and make up to the mark. One millilitre of this solution is equivalent to 1.0 mg of alumina (as Al_2O_3).

NOTE — Mercury will not dissolve and may be removed while transferring the solution to 1 000 ml volumetric flask.

D-4.2.11 Standard Zinc Solution (0.01 M) — Dissolve 5.4845 g of zinc acetate [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$] in 50 ml of water. Add 0.5 ml of glacial acetic acid and 25 ml of acetate buffer (see **D-4.2.9**) and dilute to 1 litre in a volumetric flask. Further dilute 100 ml of this solution to 250 ml in a volumetric flask and find out the equivalent Al_2O_3 , in g/ml, of this diluted solution from the procedure given in **D-4.3.3**, taking 20 ml of standard aluminium solution (see **D-4.2.10**).

D-4.2.12 Xylenol Orange Indicator — Dissolve 0.1 g of xylenol orange in 10 ml of water containing two drops of dilute hydrochloric acid (1:1).

D-4.2.13 Ammonium Fluoride — Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 3 drops of xylenol orange (see **D-4.2.12**) and then dilute hydrochloric acid (1:9) dropwise till the colour changes to yellow.

D-4.3 Procedure

D-4.3.1 Solution of the Sample

Weigh accurately about 2 g of the finely ground test sample (see 7.2) in a platinum dish. Moisten the sample with 5 ml of water. Add 3 ml of dilute sulphuric acid and 20 ml of hydrofluoric acid and evaporate slowly to dryness on the sand bath or hot plate in a fume cupboard, taking care to avoid spurting. Repeat the process with 10 ml of hydrofluoric acid. Cool, add 2 ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease to be evolved. Raise the temperature to 1 000°C and ignite for 5 min. Cool and fuse the residue with 1 g of sodium carbonate and dissolve in hydrochloric acid, cool and dilute to 250 ml in a volumetric flask.

D-4.3.2 Transfer 100 ml aliquot of the solution prepared in **D-4.3.1** to a 500 ml beaker and nearly neutralize with sodium hydroxide solution. Add with constant stirring another 100 ml of sodium hydroxide solution, containing 1 g of sodium carbonate dissolved in it. Allow to stand on a warm water bath for 1 h. Finally cool to room temperature, transfer quantitatively to a 250 ml volumetric flask and dilute up to the mark with water. Mix well and allow to stand for few minutes. Filter through a filter paper previously washed with sodium hydroxide solution, collecting the filtrate in a beaker, rinsing the beaker first with a little filtrate.

NOTE — Sodium carbonate is used for precipitating calcium and magnesium along with iron and titanium.

D-4.3.3 Transfer a suitable aliquot from the filtrate to a 250 ml conical flask, add two drops of methyl orange indicator followed by 1:1 dilute hydrochloric acid dropwise until the colour changes from yellow to red. Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina (1 ml of 0.025 M EDTA = 1.25 mg Al_2O_3 approximately). Add dilute ammonia dropwise until the colour changes from red to yellow. Add 5 ml of acetate buffer and about 25 ml water and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 5 to 6 drops of xylene orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc solution.

D-4.4 Calculation

$$\text{Alumina, percent by mass} = \frac{V \times F}{M} \times 100$$

where

V = volume of standard zinc required for the titration of liberated EDTA, in ml;

F = equivalent Al_2O_3 of standard zinc solution, in g/ml; and

M = mass of the sample represented by 100 ml of solution taken in **D-4.3.2**, in g.

D-5 DETERMINATION OF IRON OXIDE

D-5.1 Outline of the Method

The orange-red complex produced in acid solution of the sample (pH 4 to 6) by the addition of *o*-phenanthroline is determined photometrically at approximately 510 nm.

D-5.2 Reagents

D-5.2.1 Tartaric Acid Solution — 10 percent (m/v).

D-5.2.2 Hydroxylamine Hydrochloride Solution — Dissolve 1 g of the reagent in water. Transfer the solution to a 100 ml volumetric flask, dilute to the mark and mix well.

D-5.2.3 Ortho-Phenanthroline Solution — Dissolve 1 g of *o*-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

D-5.2.4 Acetate Buffer — Dissolve 21.5 g of the sodium acetate (CH_3COONa , 3 H_2O) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 000 ml.

D-5.2.5 Standard Iron Solution — Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to about 100 ml. Transfer to a 1 000 ml volumetric flask, dilute to the mark and mix well. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. One millilitre of this diluted solution is equivalent to 0.02 mg of iron (as Fe).

D-5.3 Procedure

Transfer a 5 ml aliquot of the solution prepared in **D-4.3.1** to a 100 ml volumetric flask. Also transfer 0, 1, 2, 4, 6, 8 and 10 ml each of the standard iron solution to seven separate 100 ml volumetric flasks. Then add to each flask 2 ml of tartaric acid solution followed by 2 ml of hydroxylamine hydrochloride solution. Stir well and add 5 ml of *o*-phenanthroline solution and 10 ml of acetate buffer solution. Allow to stand for 15 min and dilute up to the mark with water.

D-5.3.1 Starting with the blank (0 ml) solution transfer a suitable portion of the coloured solution, one at a time, to an absorption cell; record the photometric readings using the light band centred approximately at 510 nm, prepare the calibration curve from these readings against concentration of iron (as Fe).

D-5.3.2 Similarly transfer a suitable aliquot of the coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of iron by means of the calibration curve and calculate the iron oxide content in the aliquot taken in **D-5.3**.

D-5.4 Calculation

Ferric oxide as

$$(\text{as Fe}_2\text{O}_3), \text{ percent by mass} = \frac{M}{M_1} \times 0.1429$$

where

M = mass of iron (as Fe) found in aliquot, in mg; and

M_1 = mass of the sample represented by the aliquot taken in **D-5.3**, in g.

D-6 DETERMINATION OF TITANIUM DIOXIDE

D-6.1 Outline of the Method

The yellow coloured complex, produced in acid solution of the sample by the addition of hydrogen peroxide, is determined photometrically at approximately 410 nm.

D-6.2 Reagents

D-6.2.1 Dilute Sulphuric Acid (1:1) (v/v) — see IS 266.

D-6.2.2 Phosphoric Acid — Add 400 ml of phosphoric acid (rd 1.75) to 500 ml of water, cool and dilute to 1 litre.

D-6.2.3 Hydrogen Peroxide — 20 volumes.

D-6.2.4 Standard Titanium Dioxide Solution — Weigh accurately 3.680 g of potassium titanyl oxalate [$K_2TiO_2(C_2O_4)_2 \cdot 2H_2O$]. Transfer to a boiling flask, add 8 g of ammonium sulphate and 100 ml of concentrated sulphuric acid. Gradually heat the mixture to boiling and boil for 10 minutes. Cool, pour the solution into 750 ml of water, and dilute to 1 000 ml in a volumetric flask. One millilitre of this solution is equivalent to 0.5 mg of titanium dioxide (as TiO_2).

D-6.3 Procedure

Transfer 25 ml of the prepared solution in **D-4.3.1** to a 100 ml volumetric flask. Add 2.5 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 5 ml of hydrogen peroxide. Make up to the mark with water and mix.

D-6.3.1 Calibration Curve

Transfer 25 ml each of the test blank solution to six 100 ml volumetric flasks and add 0, 1, 2, 3, 4 and 5 ml respectively of the standard titanium dioxide solution, using the same quantity of reagents and procedure as in **D-6.3**. Make up the solutions up to the mark. Take the required aliquots of these solutions in the absorption cell, one at a time, and plot a curve showing optical densities of solutions at 410 nm against the amount of titanium dioxide in milligrams present in each.

NOTE — The test blank solution shall be prepared by using the same reagents as given in **D-4.3.1**, but without the material and dilute the solution with water to 250 ml in a volumetric flask.

D-6.3.2 Next take appropriate aliquot of the solution prepared in **D-6.3** in the absorption cell and measure its optical density at 410 nm and note from the calibration curve the amount of titanium dioxide in milligram present in it.

D-6.4 Calculation

$$\text{Titanium dioxide, percent by mass} = \frac{M_1}{M} \times 0.1$$

where

M_1 = mass of titanium dioxide content present in the aliquot taken for the test (see **D-6.3.2**), in mg; and

M = mass of the material present in the aliquot taken for the test (see **D-6.3**), in g.

D-7 DETERMINATION OF CALCIUM OXIDE

D-7.1 Outline of the Method

In an aliquot of the sample solution, metallic oxide impurities group elements (R_2O_3) are masked by triethanolamine, and calcium is titrated with EDTA using Patton and Reader (P & R) indicator.

D-7.2 Reagents

D-7.2.1 Concentrated Nitric Acid — see IS 264.

D-7.2.2 Perchloric Acid — 70 percent (m/m).

D-7.2.3 Hydrofluoric Acid — 40 percent (see IS 10332).

D-7.2.4 Concentrated Hydrochloric Acid — see IS 265.

D-7.2.5 Triethanolamine Solution — 30 percent (v/v). Dilute 60 ml of triethanolamine to 200 ml.

D-7.2.6 Sodium Hydroxide Solution (5 N approximately) — Dissolve 100 g of sodium hydroxide in water, cool and dilute to 500 ml and store in polyethylene bottle.

D-7.2.7 Patton and Reader (P & R) Indicator — Mix by grinding together 0.1 g of P & R indicator and 30 g of sodium chloride. About 0.2 g of the mixture is required for each titration.

D-7.2.8 Standard Ethylene Diamine Tetraacetic Acid (EDTA) Solution (0.01 M) — Same as **D-4.2.6**. Standardize against the standard calcium solution (see **D-7.2.9**) using P & R indicator.

D-7.2.9 Standard Calcium Solution — Dissolve 1.784 8 g of calcium carbonate dried at 150°C in a slight excess of hydrochloric acid, boil to expel carbon dioxide, cool and dilute to 1 000 ml. One millilitre of this solution is equivalent to 1 mg of calcium oxide (as CaO).

D-7.3 Procedure

D-7.3.1 Solution of the Sample

Weigh accurately about 1 g of the test sample (see **7.2**) in a platinum dish. Add 5 ml of concentrated nitric acid, 5 ml of perchloric acid and about 10 ml of hydrofluoric acid. Cover the dish with a lid. Allow the reaction to proceed for at least 15 min, then remove the lid and evaporate the mixture in dryness on a sand bath in a fume cupboard, taking care to avoid spurting. Cool,

add 5 ml of perchloric acid, rinse the lid and sides of the dish with water and again evaporate carefully to dryness. To the cool, dry residue, add 2 ml of concentrated hydrochloric acid and about 15 ml of water. Digest on a steam bath for 10 min, cool, filter if necessary and dilute the solution with deionized water to 250 ml in a volumetric flask. Store the solution in a polyethylene bottle.

D-7.3.1.1 Determination of calcium oxide

Pipette 50 ml of the solution into a 250 ml conical flask. Add 10 ml triethanolamine solution, 5 ml of sodium hydroxide solution dropwise while shaking the solution, and then add 0.2 g of P & R indicator. Titrate immediately with standard EDTA solution until the colour changes from pink to blue.

NOTE — If sharp end-point is not obtained, add 2 ml of potassium cyanide (10 percent *m/v*) after the addition of triethanolamine.

D-7.3.1.2 Calculation

$$\text{Calcium oxide, percent by mass} = \frac{V \times F}{M} \times 100$$

where

V = volume of standard EDTA solution required for titration, in ml;

F = CaO equivalent of the standard EDTA solution, in g/ml; and

M = mass of the sample represented by the aliquot taken, in g.

D-8 DETERMINATION OF ALKALIS AS POTASSIUM OXIDE AND SODIUM OXIDE

D-8.1 General

Sodium and potassium contents are determined with the help of a flame emission spectrophotometer by measuring the intensity of radiation emitted at 767 nm by potassium or at 589 nm by sodium. For this purpose use may also be made of a filter type flame photometer. In both the cases, however, the intensity of radiation either at the specified wavelength, or through the relevant filter is compared with that obtained for simulated standard solutions prepared from potassium chloride and sodium chloride solutions.

NOTE — Water used in preparing standard solutions shall be double-distilled so that it produces no deflections of the galvanometer when the instrument is set for full scale deflection for 10 ppm of potassium or 5 ppm of sodium concentration.

D-8.2 Apparatus

D-8.2.1 Flame Photometer — Provided with arrangement for monochromatic light or a set of filters required for determination of potassium and sodium. The photometer comprises essentially a galvanometer,

a photo cell or tube and a photo-multiplier tube. Other accessories are an atomizer-burner (or an atomizer and a burner) fed with liquefied petroleum gas (LPG) and compressed air (or oxygen, acetylene, etc) in such a ratio that combustion of gas is complete.

D-8.2.2 Burette — 50 ml nominal capacity and graduated in 0.1 ml for class A accuracy (*see* IS 1997).

D-8.2.3 Volumetric Flasks — Two of 1 000 ml, two of 250 ml and 14 of 100 ml nominal capacity, of class A accuracy (*see* IS 915).

D-8.3 Reagents

D-8.3.1 Hydrochloric Acid (1:1) (*v/v*) — *see* IS 265.

D-8.3.2 Standard Sodium Solutions — Weigh accurately 0.508 4 g of sodium chloride (NaCl), previously dried to constant mass at $110 \pm 2^\circ\text{C}$. Dissolve in water, transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix.

D-8.3.2.1 Transfer 10 ml of this solution to a 100 ml volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 200 ppm of sodium (as Na).

D-8.3.2.2 Transfer 25, 20, 15, 10 and 5 ml of the standard sodium solution prepared in **D-8.3.2.1** to five 100 ml volumetric flasks. Add 37.5 ml each of the potassium solution prepared in **D-8.3.3** to the five flasks, dilute up to the mark with water and mix. These solutions contain 50, 40, 30, 20 and 10 ppm respectively of sodium (as Na).

D-8.3.3 Standard Potassium Solution — Weigh accurately 0.381 4 g of potassium chloride (KCl), previously dried at $110 \pm 2^\circ\text{C}$ to constant mass. Dissolve in water, transfer to a 1 000 ml volumetric flask, dilute up to the mark with water and mix.

D-8.3.3.1 Transfer 10 ml of this solution to a 100 ml volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 200 ppm of potassium (as K).

D-8.3.3.2 Transfer 35, 30, 25, 20, 15, 10 and 5 ml of the standard potassium solution prepared in **D-8.3.3.1** to seven 100 ml volumetric flasks. Add 26.0 ml each of the sodium solution prepared in **D-8.3.2** to the seven flasks, dilute up to the mark with water and mix. One millilitre of these solutions is respectively equivalent to 70, 60, 50, 40, 30, 20 and 10 ppm of potassium (as K).

D-8.4 Setting-up the Apparatus

Switch the instrument on and wait for its stabilization. Adjust the slit and wavelengths (767 nm for potassium or 589 nm for sodium) or place the filters in position as the case may be.

D-8.4.1 Pour water in the spray cup, open its tap and turn the compressed air or oxygen on. Adjust the tap and pressure to establish atomization of water in a fine spray. Open the gas tap and light the burner. Adjust gas, if necessary, to get complete combustion. Next switch the photocell on and adjust the indicator needle or the indicator light spot, as the case may be, to bring it to the 'zero' position.

D-8.4.2 Atomize potassium (70 ppm, *see* D-8.3.3.2) or sodium (50 ppm, *see* D-8.3.2.2) solutions in place of water (*see* D-8.4.1) and adjust the deflection to full scale. Atomize water once again to check the indicator needle or light spot rests at 'zero'. If it shifts from 'zero' again adjust it to 'zero'. Thus when the instrument reads 'zero' with water and shows full scale deflection for 70 ppm K, or 50 ppm Na solution, it is ready for use.

NOTES

- 1 Atomization of any solution should always be followed by atomization of double-distilled water till the indicator comes to zero position.
- 2 Do not disturb the setting of the atomizer or burner so that rates of flow are constant for a series of determination.

D-8.5 Procedure

D-8.5.1 Preparation of the Sample Solution

Transfer 50 ml of the solution prepared in D-7.3.1 to a 500 ml volumetric flask and dilute up to the mark with water.

D-8.5.2 Calibration Graph and Determination

Without disturbing the setting of the instrument, atomize standard potassium (*see* D-8.3.3.2) or sodium (*see* D-8.3.2.2) solutions of different concentrations in an ascending order and plot the readings of the indicator against concentrations and draw the graph. Next atomize the sample solution reserved in D-8.5.1 and

note the deflection and calculate concentration of potassium or sodium as the case may be.

Example:

Suppose reading for 70 ppm K solution is 90.0, that for 60 ppm K solution is 72.0 and that for the sample solution is T (somewhere between 90.0 and 72.0).

Similarly, suppose reading for 40 ppm Na solution is 80.0, that for 50 ppm Na solution is 100.0; and that for sample solution is T (somewhere between 80.0 and 100.0).

Then concentration of potassium (C_K) in the sample solution is expressed as:

$$C_K = 60 + \frac{70 - 60}{90 - 72} \times (T - 72) \text{ ppm, or}$$

$$C_K = 70 - \frac{70 - 60}{90 - 72} \times (90 - T) \text{ ppm}$$

Similarly, concentration of sodium (C_{Na}) in the sample solution is expressed as:

$$C_{Na} = 40 + \frac{50 - 40}{100 - 80} \times (T - 80) \text{ ppm, or}$$

$$C_{Na} = 50 - \frac{50 - 40}{100 - 80} \times (100 - T) \text{ ppm}$$

D-8.6 Calculation

- a) Alkalis (as K_2O), = $1.204 C_K$, and percent by mass
- b) Alkalis (as Na_2O), = $1.348 C_{Na}$ percent by mass

where

C_K = concentration of potassium (as K) in test solution, in ppm; and

C_{Na} = concentration of sodium (as Na) in test solution, in ppm.

ANNEX E

(Clause 7.1)

SAMPLING PROCEDURE FOR SERICITE FOR CERAMIC INDUSTRY

E-1 SCALE OF SAMPLING

E-1.1 Lot

All the bags containing the material in a consignment and belonging to the same batch of manufacture and of same grade shall constitute a lot. If the consignment is known to consist of different batches of manufacture,

the bags belonging to same batch and variety shall be grouped together and each such group shall constitute a separate lot.

E-1.1.1 Unless otherwise agreed to between the buyer and the seller the number of bags to be selected shall depend upon the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Bags to be Selected

(Clause E-1.1.1)

Sl No.	No. of Bags in the Lot	No. of Bags to be Selected
(1)	(2)	(3)
i)	Up to 150	3
ii)	151-300	4
iii)	301-500	5
iv)	501-1 000	7
v)	1 001 and above	10

E-1.1.2 The bags shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS 4905 may be followed.

E-2 NUMBER OF TESTS

E-2.1 The bags shall be visually examined for defects as given in 5.1.

E-2.2 Test for moisture content and relative density shall be conducted on individual sample.

E-2.3 For fusibility and fired colour, two bags shall be selected from the bags selected as in E-1.1.1 and two individual tests shall be carried out.

E-2.4 Tests for all other characteristics in Table 1 shall be conducted on composite sample.

E-2.5 For preparation of composite sample and general requirements of sampling methods given in IS 8883 (Part 1) may be followed.

E-3 CRITERIA FOR CONFORMITY

E-3.1 For individual test, from the test results average ($\bar{X}$) and range (R) shall be calculated, range being the difference between the maximum and minimum of the test results and

$$\text{Average } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

The lot shall be declared as conforming to the specification with respect to relative density if the expression:

$$R(U - L) \leq 0.9$$

$$(\bar{X}) + 0.6 R \leq U$$

$$(\bar{X}) - 0.6 R \geq L$$

where U is upper specification limit and L is the lower specification limit.

E-3.2 Two tests of moisture content, relative density, fusibility and fired colour shall pass.

E-3.3 All the composite sample shall meet the relevant requirement given in Table 1.

E-3.4 The lot shall be declared as conforming to the requirements of this standard if the requirements E-3.1 and E-3.3 are satisfied.

NOTE — In case the number of test results in the sample is 10, they shall be divided into two subgroups of five test results each taking them consecutively obtained. The range of each subgroup then be determined and mean range shall be calculated by dividing of sum of the ranges by two. In the expression in E-3.1, R shall then be replaced by $\bar{R}$.

ANNEX F

(Foreword)

COMMITTEE COMPOSITION

Ceramicware Sectional Committee, CHD 9

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Central Glass & Ceramic Research Institute, Ahmedabad
All India Pottery Manufacturers Association, Kolkata

Central Glass & Ceramic Research Institute, Kolkata
Cera Sanitaryware Ltd, Kadi, Delhi

Chemicals & Allied Products Export Inspection Council,
Kolkata

College of Ceramic Technology, Kolkata

Controller of Quality Assurance, Kanpur

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SHRI GIRISH TRIVEDI (*Alternate*)

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